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JC986 U.S. PTO

ARM PTO-1082

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Washington, D.C. 20231

EXPRESS MAIL NO. EL584667316US  
Case Docket No. 57974-5002  
Date: November 3, 2000

1c931 U.S. PTO  
09/706158  
11/03/00

Sir:

Transmitted herewith for filing is the Continuation-In-Part patent application under CFR §1.53(b)(2) of:

Inventor(s): TOR McPARTLAND

For: ANT SPRAY CONTAINING D-LIMONENE AND METHODS OF MAKING AND USING SAME

Enclosed are:

-0- Sheet(s) of drawing ( ☐ formal ☒ informal ) +      extra copies

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An assignment of the invention to \_\_\_\_\_  
Will follow.  
  
A certified copy of \_\_\_\_\_  
from which priority is claimed in the subject case pursuant to Rule 55(b) and 35 USC 119(a)-(d).  
Will follow.  
  
A Power of Attorney by Assignee and Exclusion of Inventor Under Rule 3.71.  
A Verified Statement to Establish Small Entity Status under 37 CFR 1.9 and 37 CFR 1.27.  
Declaration - Unsigned  
Recordation Form Cover Letter (Form PTO 1595)  
Information Disclosure Statement (Form PTO 1449), with 5 attachments  
Petition to Make Special under 37 CFR §1.102 and MPEP §708.02(VIII).  
Petition for Extension of Time - 3 months. Applicant paid for 2 months.

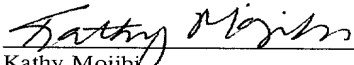
FOR:	NO FILED	NO EXTRA	SMALL ENTITY		OR	OTHER THAN SMALL ENTITY	
			RATE	FEE		RATE	FEE
BASIC FEE				\$ 355			\$ 710
TOTAL CLAIMS	26 - 20	6	x 9 =	\$ 36	OR	x 18 =	\$
INDEP CLAIMS	5 - 3	2	x 40 =	\$ 80	OR	x 80 =	\$
MULTIPLE DEPENDENT CLAIMS PRESENTED			+ \$135			+\$270	
TOTAL				\$ 471			\$

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Please charge my Deposit Account No. 10-0440 the amount of \$\_\_\_\_\_.  
A check in the amount of \$ 605 to cover the filing fee is enclosed. A check in the amount of \$258 was submitted with the Amendment filed on October 3, 2000; the Amendment was not entered. Accordingly, the fee should be credited to Deposit Account No. 10-0440 A duplicate copy of this sheet is enclosed.  
Check for \$40.00 covering Recordation of Assignment fee enclosed.  
The Commissioner is hereby authorized to charge payment of the following fees associated with this communication or credit any overpayment to Deposit Account No. 10-0440. A duplicate copy of this sheet is enclosed.  
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☐ The issue fee set in 37 CFR 1.18 at or before mailing of the Notice of Allowance, pursuant to 37 CFR 1.311(b).  
☒ Any filing fees under 37 CFR 1.16 for presentation of extra claims.

Respectfully submitted,

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Rev. 06/1/94

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PATENT  
57974-5002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of:  
Tor McPartland

Serial No. UNASSIGNED

Filed: HEREWITH

For: ANT SPRAY CONTAINING D-  
LIMONENE AND METHODS OF  
MAKING AND USING SAME

Group Art Unit:

Examiner:



CERTIFICATE OF MAILING

Box Patent Application  
Assistant Commissioner for Patents  
Washington, D.C. 20231

I hereby certify that a Continuation-In-Part Patent Application Under 37 CFR 1.53(b)(2) including fourteen (14) pages of Specification; Application Transmittal Form PTO-1082 (duplicate); Check No. 1718 in the amount of \$605; Declaration (unsigned); Information Disclosure Statement with 5 cited references; and return postcard are being deposited with the United States Postal Service, postage prepaid, Express Mail No. EL584667316US on the date indicated below and addressed to: Box Patent Application, Assistant Commissioner for Patents, Washington, D.C. 20231.

November 3, 2000

(Date of Deposit)

Rebecca Sargent

(Name of Person Mailing Paper or Fee)

*Rebecca Sargent*

(Signature)

**APPLICATION FOR  
UNITED STATES PATENT  
IN THE NAME OF**

**TOR McPARTLAND**

**FOR**

**ANT SPRAY CONTAININT D-LIMONENE  
AND METHODS OF MAKING AND USING SAME**

**DOCKET NO. 57974-5002**

***Prepared by***

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PATENT  
57974-5002

**ANT SPRAY CONTAINING D-LIMONENE  
AND METHODS OF MAKING AND USING SAME**

This is a continuation-in-part of U.S. Patent Application Serial No. 09/218,732, filed December 22, 1998, which is a continuation-in-part of U.S. Patent Application Serial No. 08/846,351, filed April 30, 1997, both of which are now abandoned and the disclosures of which are incorporated in their entirety herein by reference.

Field of the Invention

The present invention relates to an *all-natural, food grade* insecticidal composition that is effective in controlling insects including ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body and head lice and more particularly relates to an insecticidal composition that contains D-limonene, a non-toxic hydrophilic solvent, a non-toxic emulsifying agent, and a preservative, that is *an effective residual repellent and contact insecticide* but is non-toxic to humans and household animals, and not harmful to landscaping, particularly rose bushes and ornamentals, indoor plants or the environment.

Background of the Invention

Numerous pesticide and insecticide products are available in the market for killing insects such as ants. However, these products are generally toxic to humans and household animals, and harmful to plants and the environment. Therefore they must be administered with extreme care. This is especially a problem in the termination of ants, cockroaches and the like because they normally appear near food where a careless use of insecticides may cause accidental poisoning of humans and household animals.

Other insecticide products containing D-Limonene disperse the D-Limonene in mineral oil petroleum distillates which may be harmful if accidentally ingested and

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may cause damage to plants. Unlike the compositions of the present invention, no emulsifier is used because the D-Limonene is dispersed in oil and not water.

Lice infestation of humans, particularly children, is still treated with compositions that contain the pesticide lindane. Lindane *is* carcinogenic **and an estrogen mimic**. There exists a need for a composition which can treat body and head lice but which is safe to use, particularly on children.

U.S. Patent No. 4,379,168 to Dotolo discloses pesticides containing D-limonene as an insect-killing ingredient along with water-soluble surfactants or emulsifiers, and water. The pesticide compositions are designed for use mainly as a dip to rid small animals of fleas and ticks and as a spray to kill fleas and ticks on small animals. None of the compositions taught by Dotolo contain suitable amounts of emulsifying agent and D-limonene for the purpose of the present invention. None of the compositions taught by Dotolo contain any surfactants or emulsifiers that were selected for their safety. For example, the Kodak Laboratory Chemical Catalog No. 51 indicates that Triton X-100, which is disclosed as an acceptable emulsifier in Dotolo, is irritating to the skin and eyes.

U.S. Patent No. 3,023,144 to Greathouse, et al. discloses germicides and fungicides containing about 25% by weight D-limonene, about 1% by weight of p-methyl acetophenone, and other unsaturated hydrocarbon cleavage products of D-limonene, up to about 7% by weight concentrated citrus oil foots and from 0.25% up to about 10% by weight salicylic acid. The compositions are used for topical application on humans and animals to control infections of skin and external organs arising from wounds or from infestation by fungi, bacteria, and larvae. Greathouse discloses that the active ingredient for the biocidal activity of the compositions disclosed is not D-limonene but rather compounds such as p-methyl acetophenone, and other unsaturated hydrocarbon cleavage products of D-limonene.

A need exists for an insecticidal composition that ***kills on contact and*** controls insects such as ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body

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and head lice by repelling them, and which is non-toxic to humans, household animals and house plants.

A need also exists for an insecticidal composition that is effective in controlling insects such as ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, ***spiders, earwigs, slugs and snails***, cockroaches, flies, wasps, body and head lice by killing them, and which is non-toxic to humans, household animals and house plants.

There is also a need for a safe and effective treatment for lice infestation of a human.

A need also exists for an insecticidal composition that contains an insecticide made from a natural substance that is part of and therefore not harmful to landscaping, particularly rose bushes and ornamentals, indoor plants or the environment.

***A need also exists for an insecticidal composition that can be used around food, humans and pets without worry.***

#### Summary of the Preferred Embodiments

In accordance with one aspect of the present invention, there is provided a food-grade insecticidal composition that includes an amount of D-limonene sufficient to provide insect control, a non-toxic hydrophilic solvent, and an amount of a non-toxic emulsifying agent sufficient to solubilize D-limonene in the solvent.

Preferably, the inventive composition includes about 1% to about 20% by weight of D-limonene, about 1% to about 25% by weight of the selected non-toxic emulsifying agent, for example Alkamuls EL620, and about 98% to about 55% by weight of the selected non-toxic hydrophilic solvent.

In a preferred embodiment, the inventive composition also includes at least one food-grade preservative, such as sodium benzoate.

Methods of making and using the inventive compositions are also provided.

Other objects, features and advantages of the present invention will become apparent to those skilled in the art from the following detailed description. It is to be understood, however, that the detailed description and specific examples, while

indicating preferred embodiments of the present invention, are given by way of illustration and not limitation. Many changes and modifications within the scope of the present invention may be made without departing from the spirit thereof, and the invention includes all such modifications.

#### Detailed Description of the Preferred Embodiments

The non-toxic insecticidal compositions according to the present invention have a pleasant citrus scent and are suitable for use in any living environment. In particular, the inventive compositions are made only from food-grade ingredients. As used herein, an ingredient or a composition is "food-grade" if it meets the U.S.F.D.A.'s G.R.A.S. (Generally Recognized as Safe) food grade standard. Additionally, in a preferred embodiment of the invention, the inventive compositions are made only from organic ingredients. As used herein, an ingredient or a composition is "organic" if it is made mainly of naturally occurring materials. Thus, the inventive compositions are not only safe and non-toxic to humans and household animals such as dogs, cats, rabbits, ***birds, lizards***, etc., but can also be used near food without any danger of contamination or accidental poisoning, and are safe even if ingested by humans or household animals. The inventive compositions will not harm landscaping foliage or indoor plants. Since the inventive compositions are made of mainly naturally occurring materials, they are not harmful to the environment and do not cause any unwanted pollution. They are also completely biodegradable.

D-limonene, otherwise known as orange limonene or 1-methyl-4-(1 methylethenyl) cyclohexene or 4-isopropenyl-1-methyl cyclohexene, has a chemical formula of  $C_{10}H_{16}$ , a molecular weight of 136.2, and contains 88.1% C and 11.8% H by weight. It occurs in various ethereal oils, particularly in oils of lemon, orange, lime, grapefruit and bergamot. D-limonene can be obtained from steam extraction of citrus peels of orange, lemon, lime, grapefruit and bergamot, some of the extractions can contain as high as 90% D-limonene. The invention thus provides a valuable use for what would otherwise be a waste product. Distillation of the oils produces technical grades of D-limonene of higher purity, *i.e.*, from about 95% to about 96%.

D-limonene has a pleasant citrus scent. It can be suitably used in any living environment.

The D-form of limonene is a liquid having a boiling point of 175.5-176 degrees centigrade. It can be commercially obtained from Lykes Pasco Packing Company (Dade City, Florida) or Florida Chemical Company (Lake Alfred, Florida).

The compositions of the present invention allow a user to provide insect control in interior and exterior settings. Insect control can include repelling and/or killing insects, such that less insects are alive or present in a given area than if the compositions of the present invention had not been applied in the area. The inventive compositions control a wide range of insects, including without limitations ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body lice and head lice.

While not limiting the invention by any particular theory, it is believed that the D-limonene acts to soften the waxy coating on the exoskeleton of insects and thereby causes the softened coating to clog the external insect respiratory organs, known as spiracles. The clogged spiracles interfere with the ability of the insect to obtain adequate amounts of oxygen, ultimately resulting in the death of the insect. This invention is therefore also suitable against other pests, besides insects, which would be susceptible to the external effects set forth above.

The inventive compositions include a food-grade, non-toxic hydrophilic solvent. Water is particularly preferred as the solvent. Other non-toxic hydrophilic solvents, for example, ethanol, dilute acetic acid solutions, and the like can also be used.

Useful emulsifying agents include polyethoxylated castor oils. One such emulsifying agent is available commercially under the trade name of Alkamuls EL620 from Rhone Poulenc Co. It is non-toxic to humans, household animals and house plants and landscaping and will not cause skin or eye irritation. Castor oils also improve the insecticidal effect of the formulation. Other commercially available emulsifying agents that are non-toxic, such as polyoxyethylenesorbitans supplied by ICI Americas or Sigma Chemical Company, may also be suitably used for the present

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invention provided that they are food-grade. In a preferred embodiment a polyoxyethylenesorbitan monooleate such as Tween 80 may be used.

In general, the emulsifying agent should be present in an amount sufficient to render the D-limonene soluble in the non-toxic hydrophilic solvent. When a polyethoxylated castor oil is used, it should contain sufficient polyethoxylation to render the D-limonene soluble in a non-toxic hydrophilic solvent when the emulsifying agent is used in an amount as disclosed herein. ***When that solvent is water, the temperature of the water when combined with the other ingredients of the composition should be about 95 °F for optimal emulsification.***

In order to provide a reasonable shelf-life to the insecticidal compositions, it is preferable that a preservative be added to the composition. One such suitable preservative is sodium benzoate commercially supplied by Pfizer, Inc. Other commercially available preservatives used for preserving food, as would be known to those of ordinary skill in the art, may also be suitably used.

Preferred embodiments of the inventive insecticidal composition, which are suitable for application as a spray, include about 0.7% to about 20% by weight, more preferably about 0.7% to about 10% by weight, of D-limonene, and even more preferably about 0.775% by weight D-limonene; between about 1% to about 25% by weight, more preferably about 5% to about 15% by weight, of a non-toxic emulsifying agent; about 0.01% to about 5% by weight, more preferably about 0.01% to about 1.0% by weight, of a preservative; and the balance of the selected solvent.

Preferably, the inventive compositions are free of petroleum distillates.

When the novel insecticidal compositions are used indoors as a spray, they should be sprayed preferably from about 6 to about 8 inches away from and directly on insects such as ants, or on insect trails such as ant trails to the source of the insects such as ants (e.g., ant colonies, nests, etc.) and sprayed until visibly wet. Insects are typically killed within minutes of contact with the novel insecticidal compositions.

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The insecticidal compositions can be used on the interior surfaces in a building such as counter tops and in food preparation areas. For outdoor use, the insecticidal compositions of the present invention should be applied at the perimeter of a building such as a home, at insect trails such as ant trails, at insect nests, such as ant nests and at doors, cracks, and window frames where insects such as ants may enter the building. It should be applied until visibly wet. It will provide lasting repellent qualities.

Application of the present insecticidal compositions is preferably effected by spraying of the insecticidal compositions by conventional spray apparatus such as aerosol cans bug sprayers and the like. However, application may also be effected by any means of contacting surfaces to be treated, for example, with a brush which has been dipped in the insecticidal compositions. Because the novel insecticidal compositions are non-toxic, they can also be applied with a human hand.

When the present invention is applied to solid surfaces and left to remain there, the residual effect of the insecticidal composition will last for a period of time effectively keeping insects, such as ants, away from the treated area.

The inventive insecticidal compositions can also be applied to a plant, such as a rose bush or other ornamental plant, in order to control insects.

Further embodiments of the inventive compositions can be formulated for use in treating humans, particularly children, infected with lice, particularly head lice. When the novel insecticidal composition is used to treat humans infected with lice, it should be applied to the infected area, such as the scalp and left on for about 5 minutes after which it may be rinsed and preferably shampooed off.

The invention can be made as a concentrate that can be diluted with water. The invention can be used as a paste and be effective in the control of head and body lice.

*Non-limiting examples of the inventive insecticidal composition are set forth below.*

Example 1

*For home, office, school, institutional or industrial applications, indoor and outdoor (proven effective against ants, roaches and fleas but is a broad based insecticide and repellent):*

6 wt% D-limonene (**0.2% impurities in the D-limonene, net active 5.8%**)  
10 wt% Alkamuls EL620 (polyethoxylated caster oil)  
0.1 wt% sodium benzoate  
**83.9** wt% water.

The insecticidal composition according to the foregoing specific formulation has a white, opalescent color. Its physical state is a liquid at 25 °C and has a citrus-like odor. It has a boiling point between 99-100°C and a specific gravity of 0.9753 gm/ml. It is soluble in water and has a pH of 6.1. The flash point of the insecticidal composition is greater than 60°C. It has a viscosity of 1.97 centipoise at 37.8°C.

Example 2

For agriculture, on food and ornamental crops (proven effective on aphids and spider mites but useful for many other plan pests):

0.775% D-limonene (.05% impurities in the d-Limonene, net active 0.725%)  
2.5% Alkamuls EL 620  
0.025% Sodium Benzoate  
95.975% Water (UV sterilized and particulate filtered to 5 microns)

Example 3

As a liquid concentrate to make the two previous formulations by simply adding the proper ratio of warm 95°F water:

18% d-Limonene (**0.6%** impurities in the d-Limonene, net active **17.4%**)  
30% Alkamuls EL 620  
0.3% Sodium Benzoate  
51.7% Water (UV sterilized and particulate filtered to 5 microns)

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Example 4

As a paste for applying to head and body as a treatment for lice (puts lice into a state of morbidity making for easy removal):

24% d-Limonene (**0.8%** impurities in the d-Limonene, net active **23.2%**)

40% Alkamuls EL 620

0.1% Sodium Benzoate

35.9% Water (UV sterilized and particulate filtered to 5 microns)

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What is claimed is:

1. A food-grade insecticidal composition comprising an amount of D-limonene sufficient to provide insect control, a non-toxic hydrophilic solvent, an amount of a non-toxic emulsifying agent sufficient to solubilize D-limonene in said non-toxic hydrophilic solvent, wherein the emulsifying agent is non-toxic to humans.

2. The insecticidal composition according to claim 1 comprising about 1% to about 20% by weight of D-limonene, about 1% to about 25% by weight said non-toxic emulsifying agent, and about 98% to about 55% by weight of said non-toxic hydrophilic solvent.

3. The insecticidal composition according to claim 1 further comprising an amount of at least one food-grade preservative sufficient to extend the effectiveness of said insecticidal composition.

4. The insecticidal composition according to claim 3 comprising about 0.01% to about 5% by weight of said preservative.

5. The insecticidal composition according to claim 1 wherein said amount of D-limonene sufficient to provide insect control is sufficient to repel an insect.

6. The insecticidal composition according to claim 1 wherein said amount of D-limonene sufficient to provide insect control is sufficient to kill an insect.

7. The insecticidal composition according to claim 1 wherein said non-toxic emulsifying agent is a polyethoxylated castor oil.

8. The insecticidal composition according to claim 1 wherein said non-toxic emulsifying agent is a polyoxyethylenesorbitan.

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9. The insecticidal composition according to claim 1 wherein said non-toxic hydrophilic solvent is water.

10. The insecticidal composition according to claim 2 wherein said preservative is a food preservative.

11. The insecticidal composition according to claim 1 which controls at least one insect selected from the group consisting of ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body lice and head lice.

12. The insecticidal composition according to claim 1 comprising about 1% to about 10% by weight of D-limonene, about 5% to about 15% by weight of a non-toxic emulsifying agent, and about 94% to about 75% by weight of a non-toxic hydrophilic solvent.

13. A method of making an insecticidal composition comprising the step of combining an amount of D-limonene sufficient to provide insect control with a non-toxic hydrophilic solvent and an amount of a non-toxic emulsifying agent sufficient to solubilize D-limonene in said non-toxic hydrophilic solvent, wherein all said ingredients are food-grade.

14. The method of claim 13 comprising the step of combining about 1% to about 20% by weight of D-limonene, about 1% to about 25% by weight of said non-toxic emulsifying agent and about 97.99% to about 50% by weight of said non-toxic hydrophilic solvent.

15. The method of claim 14 wherein said solvent is water.

16. The method of claim 14 wherein said ingredients are combined with about 0.01% to about 5% by weight of a preservative.

17. A method of controlling insects comprising the step of applying the insecticidal composition of claim 1 to one selected from the group consisting of an insect, an insect trail, an insect nest, a building surface, a building perimeter, and a plant.

18. The method of claim 17 wherein said insects to be controlled are on plants.

19. The method of claim 18 wherein said plants are selected from the group consisting of rose bushes and ornamentals.

20. A method of treating a human infected with lice comprising the steps of applying the composition of claim 1 to a portion of the human infected with the lice, allowing said composition to remain on the infected portion for a period of time and removing said composition.

21. A food-grade insecticidal composition comprising about 0.7% to about 1.5% by weight of D-limonene, about 1% to about 25% by weight of a castor oil and about 98% to about 55% by weight of a non-toxic hydrophilic solvent.

22. The insecticidal composition according to claim 21 further comprising an amount of at least one food-grade preservative sufficient to extend the effectiveness of the insecticidal composition.

23. The insecticidal composition according to claim 22 comprising about 0.01% to about 5% by weight of said preservative.

24. The insecticidal composition according to claim 21 comprising about 0.775% D-limonene.

25. A method of eradicating fire ants, comprising:

applying a formulation to an area to be eradicated of fire ants, wherein said formulation consists essentially of about 0.7% to about 1.5% D-limonene, about 1% to about 25% by weight of an emulsifying agent, and about 98% to about 55% by weight of a non-toxic hydrophilic solvent; and

allowing said formulation to remain in contact with said area for a suitable period of time to eradicate said fire ants.

26. A method of eradicating fire ants, comprising:

applying a formulation to an area to be eradicated of fire ants, wherein said formulation consists essentially of about 0.7% to about 1.5% D-limonene, about 1% to about 25% by weight of an emulsifying agent, about 98% to about 55% by weight of a non-toxic hydrophilic solvent, and about 0.01% to about 5% by weight of a food-grade preservative ; and

allowing said formulation to remain in contact with said area for a suitable period of time to eradicate said fire ants.





**DECLARATION**

☐ ORIGINAL  
☒ CONTINUATION-IN-PART  
☐ DIVISIONAL

As a below named inventor, I declare that the information given herein is true, that I believe that I am the original, first and sole inventor if only one name is listed at 1 below, or a joint inventor if plural inventors are named below at 1-4, of the invention entitled:  
**ANT SPRAY CONTAINING E-LIMONENE AND METHODS OF MAKING AND USING SAME**

Which is described and claimed in:

- ☒ the attached specification or  
☐ the specification in application Serial No. \_\_\_\_\_ filed \_\_\_\_\_ ☐ as amended on \_\_\_\_\_ (if applicable)  
(for declaration not accompanying application)

and for which a patent is sought, and that my residence, post office address and citizenship are as stated below next to my name.  
I acknowledge my duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations §1.56(a).  
I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.  
I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATION(S)			
COUNTRY	APPLICATION NUMBER	DATE OF FILING (day/month/year)	PRIORITY CLAIMED UNDER 35 UCS §119
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below:

APPLICATION SERIAL NO.	FILING DATE

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code §120, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NO.	FILING DATE	STATUS
09/218,732	December 22, 1998	Pending

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